भारतीय मानक
Indian Standard

IS 10151 : 2019

खाद्य सामग्री, औषधियों एवं पेयजल के संपर्क में इनके सुरक्षित उपयोग के लिए पोलिविनायईल क्लोराइड (पीवीसी) एवं उसके कोपॉलिमर — विशिष्टि

(पहला पुनरीक्षण)

Polyvinyl Chloride (PVC) and its Copolymers for its Safe Use in Contact with Foodstuffs, Pharmaceuticals and Drinking Water — Specification

(First Revision)

ICS 67.250; 83.080.20

भारतीय मानक ब्यूरो
BUREAU OF INDIAN STANDARDS
मानक भवन, 9 बहादुरशाह ज़फर मार्ग, नई दिल्ली-110002
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI-110002
www.bis.gov.in www.standardsbis.in

March 2019 Price Group 6

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Plastics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Plastics are now being used on a large scale for packaging of foodstuffs and pharmaceuticals. Where direct contact occurs between the packed commodity and the plastics, the high molecular mass polymer itself does not pose a toxic hazard being inert and essentially insoluble in food. There is, however, a likelihood that some transfer will occur of polymer additives, adventitious impurities, such as monomers, catalystremnants and residual polymerization solvents and of low molecular mass polymer fractions from the plastics into the packaged material with consequent toxic hazard to the consumers. The occurrence of acute toxicity due to plastics materials in contact with food is most unlikely, since only trace quantities of potentially toxic materials are likely to migrate. However, the accumulation of these toxic materials with time may lead to hazards which may be serious.

This standard was originally published in 1982 and was amended from time-to-time. In this revision, all the four amendments issued so far have been incorporated. Further the requirement of residual vinyl chloride content in resin has been modified.

This standard is intended to be used with a series of Indian Standards on plastics for food contact applications which are given in Annex E. It is emphasized that these standards need to be used in combination to provide a system of control to the manufacturers of plastics as well as the fabricators of thermoplastics packaging materials to derive maximum benefit besides, it may also serve as basis for official agencies to frame suitable legislation to ensure effective safeguards for the safety and health of consumers where thermoplastics for food contact and related applications are concerned.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

POLYVINYL CHLORIDE (PVC) AND ITS COPOLYMERS FOR ITS SAFE USE IN CONTACT WITH FOODSTUFFS, PHARMACEUTICALS AND DRINKING WATER — SPECIFICATION

(First Revision)

1 SCOPE

1.1 This standard specifies the requirements and methods of sampling and test for polyvinyl chloride (PVC) and its copolymers for the manufacture of plastic items used in contact with foodstuffs, pharmaceuticals and drinking water.

1.2 This standard does not purport to establish the suitability of the packaging media with particular foodstuff, pharmaceutical and drinking water, from other than toxicological considerations.

2 REFERENCES

The following standards contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standard indicated below.

IS No.	*Title*
4905 : 2015	Random sampling and randomization procedures (*first revision*)
9833 : 2014	List of pigments and colorants for use in plastics in contact with food stuffs, pharmaceuticals and drinking water (*first revision*)
9845 : 1998	Method of analysis for the determination of specific and/or overall migration of constituents of plastics materials and articles intended to come into contact with foodstuffs (*second revision*)
10148 : 1982	Positive list of constituents of polyvinyl chloride (PVC) and its copolymers in contact with foodstuffs, pharmaceuticals and drinking water

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions of PVC given in **2** of IS l0148 shall apply.

4 REQUIREMENTS

4.1 Material

The material shall comply with the threshold limits of the catalysts, polymerization inhibitors, emulsifying agents, suspension agents, chain-transfer agents and miscellaneous additives as prescribed in IS 10148.

4.2 In case auxiliary items, that is, plasticizers, stabilizers, lubricants and other additives are used in PVC compounds for food-packaging applications it shall comply with the threshold limits as prescribed in IS 10148.

4.3 Pigments and Colourants

In case the coloured material is used for food-packaging applications, it shall comply with the list and limits of the pigments and colourants prescribed in IS 9833.

4.4 Monomer Content

4.4.1 *Residual Vinyl Chloride Monomer (RVCM) Content*

The RVCM content of PVC suspension resin used for the manufacture and PVC containers/film used for food packaging shall not exceed 1 ppm, when tested as prescribed in Annex A.

4.4.2 *Migration of RVCM into Foodstuffs*

The migration of RVCM into foodstuffs being packed shall not exceed 10 ppb when tested as prescribed in Annex B.

NOTE — In accordance with the EEC Council Directive 78/142/EEC, determination by simulants is permissible.

4.5 Overall Migration

The material shall comply with the overall migration limits of 60 mg/l, maximum of simulants and 10 mg/dm^2, maximum of the surface of the material or article when tested by the method prescribed in IS 9845.

4.5.1 *Preparation of Film/Sheet*

For overall migration determination of resin film/sheet

may be prepared using the recipe and compounding procedure given in Annex C.

4.6 Storage and Control

4.6.1 *Storage*

Plastics materials intended for food contact use shall be stored separately from other materials in closed, properly identified containers.

4.6.2 *Control*

An authorised person shall supervise and control the issue of plastics materials to the process or manufacturing area and shall maintain appropriate written records of the issue of such materials.

4.6.3 Adequate standards of hygiene shall be maintained at all times and plant operators and store men shall be trained in proper hygiene practices.

5 PACKING AND MARKING

5.1 Packing

The material shall be suitably packed with suitable liner in gunny paper bags, as agreed to between the purchaser and the supplier, in a manner so as to ensure that the items do not become contaminated during storage.

5.2 Marking

Each package shall be clearly marked with the following information:

a) Name and type of the material;
b) Net mass in the package;
c) Indication of the source of manufacturer;
d) Recognized trade-mark, if any;
e) Date of manufacture; and
f) Batch No. or Code No.

5.3 BIS Certification Marking

The container may also be marked with the Standard Mark.

5.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made there under. The details of conditions under which the license for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 The method of preparing representative test samples of the material and the criteria for conformity shall be as prescribed in Annex D.

ANNEX A

(*Clause* 4.4.1)

DETERMINATION OF RESIDUAL VINYL CHLORIDE MONOMER IN BASIC MATERIALS AND ARTICLES

A-1 OUTLINE OF THE METHOD

In this method the vinyl chloride monomer level in basic materials or articles is determined by means of gas chromatography using the 'headspace' method after dissolution or suspension of the sample in N, N-dimethylacetamide.

A-2 REAGENTS

A-2.1 Vinyl Chloride (VC), of purity greater than 99.5 percent (*v/v*).

A-2.2 N, N-dimethylacetamide (DMA), free from any impurity with the same retention time as VC or as the internal standard (**A-2.3**) under the conditions of the test.

A-2.3 Diethyl Ether or cis-2-butene, in DMA (**A-2.2**) as the internal standard solution. These internal standards shall not contain any impurity with the same retention time as VC, under the conditions of the test.

A-3 APPARATUS

A-3.1 Gas Chromatograph — Fitted with automatic head space sampler or with facilities for manual sample injection.

A-3.2 Flame Ionization Detector or Other Detectors, mentioned in **A-7**

A-3.3 Gas Chromatographic Column

The column shall permit these paration of peaks of air, or VC and of the internal standard, if used. Furthermore, the combined **A-3.2** and **A-3.3** system shall allow the signal obtained with a solution containing 0.02 mg VC/litre DMA or 0.02 mg VC/kg DMA to be equal to at least five times the background noise.

A-3.4 Sample Phials or Flasks Fitted with Silicon or Butyl Rubber Septa

When using manual sampling techniques the taking of a sample from the headspace with a syringe may cause a partial vacuum to for minside the phial or flask. Hence, for manual techniques where the phials are not pressurized before the sample is taken, the use of large phials is recommended.

A-3.5 Micro-syringes

A-3.6 Gas-Tight Syringes for Manual Headspace Sampling

A-3.7 Analytical Balance — accurate to 0.1 mg.

A-3.8 Headspace Autosampler

Temperature of the thermostat	—	180°C
Thermostating time	—	10 min
Injection time	—	0.04 min
Volume	—	250 µl
Transfer line temperature	—	200°C

A-4 PROCEDURE

CAUTION — VC is a hazardous substance and a gas at ambient temperature, therefore, the preparation of solutions should be carried out in a well ventilated fume cupboard. Take all the necessary precautions to ensure that no VC or DMA is lost.

NOTES

1 When employing manual sampling techniques an internal standard (**A-2.3**) should be used.

2 When using an internal standard, the same solution shall be utilized throughout the procedure.

A-4.1 Preparation of Concentrated Standard VC Solution at Approximately 2 000 mg/kg

Accurately weigh to the nearest 0.1 mg a suitable glass vessel and place in it a 50 ml quantity of DMA. Reweigh. Add to the DMA 0.1 g of VC in liquid or gas form, injecting it slowly onto the DMA. The VC may also be added by bubbling it into the DMA, provided that a device is used which shall prevent loss of DMA. Re-weigh to the nearest 0.1 mg. Wait for 2 h to allow equilibrium to be attained. Keep the standard solution in refrigerator.

A-4.2 Preparation of Dilute Standard VC Solution

Take a weighed amount of concentrated standard solution of VC (**A-4.1**) and dilute, to a known volume or a known weight, with DMA or with internal standard solution (**A-2.3**). The concentration of the result and dilute standard solution is expressed as mg/l or mg/kg respectively.

A-4.3 Preparation of Calibration Curve

NOTE — The curve shall comprise at least 7 pairs of points.

A-4.3.1 The repeatability of the responses shall be lower than 0.02 mg VC/l or mg VC/kg of DMA. The curve shall be calculated from these points by the least squares technique, that is, the regression line shall be calculated using the following equation:

$y = a_1 x + a_0$

where

$$a_1 = \frac{n\sum xy - \left(\sum x\right).\left(\sum y\right)}{n\sum x^2 - \left(\sum x\right)^2}$$

and

$$a_0 = \frac{\left(\sum y\right)\left(\sum x^2\right) - \left(\sum x\right).\left(\sum xy\right)}{n\sum x^2 - \left(\sum x\right)^2}$$

where

y = the height or area of peaks in any single determination;

x = the corresponding concentration on the regression line; and

n = number of determinations carried out ($n \geq 14$).

A-4.3.2 The curve shall be linear, that is, the standard deviation(s) of the differences between the measured responses (y_1) and the corresponding value of the responses calculated from the regression line (z_1) divided by the mean value ($\bar{y}$) of all the measured responses shall not exceed 0.07. This shall be calculated from:

$$\frac{s}{\bar{y}} \leq 0.07$$

where

$$S = \sqrt{\frac{\sum_{i=1}^{n}\left(y_i - z_i\right)^2}{n-1}}$$

$$\bar{y} = \frac{1}{n}\sum_{i=1}^{n} y_i$$

where

y_i = each individual measured response;

z_i = the corresponding value of the response (y_i) on the calculated regression line, and $n \geq 14$.

A-4.3.3 Prepare two series of at least 7 phials (**A-3.4**). Add to each phial volumes of dilute standard VC solution (**A-4.2**) and DMA (**A-2.3**) or internal standard solution in DMA (**A-2.3**) such that the final VC concentration of the duplicate solutions shall be approximately equal to 0, 0.050, 0.075, 0.100, 0.125, 0.150, 0.200 mg/l or mg/kg of DMA and that all the phials contain the same quantity of DMA that is to be used under **A-4.4**. Seal the phials and proceed as described under **A-4.5**. Construct a graph in which the ordinate values show the areas (or heights) of the VC peaks of the duplicate solutions or the ratio of these areas (or heights) to those of the relevant internal standard peaks and the abscissa values show the VC concentrations of the duplicate solutions. IS concentration to be maintained any where between the calibration range.

A-4.4 Validation of Preparation of Standard Solution Obtained in A-4.1 and A-4.2

Repeat the procedure described under **A-4.1**and **A-4.2** to obtain a second diluted standard solution with a concentration equal to 0.1 mg VC/l or 0.1mg VC/kg of DMA or internal standard solution. The average of two gas-chromatographic determinations of this solution shall not differ by more than 5 percent from the corresponding point of the calibration curve. If the difference is greater than 5 percent, reject all the solutions obtained in **A-4.1, A-4.2, A-4.3** and **A-4.4** and repeat the procedure from the beginning.

A-4.5 Preparation of the Samples of Materials or Articles

Prepare two phials (**A-3.4**). Weigh into each phial not less than 200 mg, to the nearest 0.1 mg, of the sample obtained from a single material or article under investigation which has been reduced to small pieces. Try to ensure that an equal quantity is weighed into each phial. Close the phial immediately. Add to each phial for each gram of the sample10 ml or 10 g of DMA (**A-2.3**) or 10 ml or 10 g of internal standard solution (**A-2.3**). Seal the phials and proceed as described under **A-5**.

A-5 GAS-CHROMATOGRAPHIC DETERMINATIONS

A-5.1 Agitate the phials avoiding contact between the contained liquid and the septum (**A-3.4**) to obtain a solution or suspension of the samples of material or article (**A-4.5**) as homogeneous as possible.

A-5.2 Put all the sealed phials (**A-4.3, A-4.4** and **A-4.5**) in a water bath for 2 h at 60 ± 1°C to allow equilibrium to be attained. Agitate again if necessary.

A-5.3 Take a sample from the headspace in the phial. When utilising manual sampling techniques care shall be exercised in obtaining are producible sample (**A-3.4**), in particular the syringe shall be pre-warmed to the temperature of the sample. Measure the area (or height) of the peaks relating to the VC and to the internal standard, if used.

A-5.4 Remove from the column (**A-3.3**) excess DMA using an appropriate method as soon as peaks of DMA appear on the chromatogram.

A-6 CALCULATION OF THE RESULTS

A-6.1 Find by interpolation on the curve, the unknown concentration of each of the two solutions of the sample taking account of the internal standard solution, if used.

Calculate the amount of VC in each of the two samples of material or article under investigation by applying the following formula:

$$X = \frac{C \quad V \quad 1\,000}{M}$$

where

X = concentration of VC in the sample of the material or article expressed in mg/kg;

C = concentration of VC in the phial containing the sample of material or article *(***A-4.5**) expressed in mg/l or mg/kg;

V = volume or mass of DMA in the phial containing the sample of material or article *(***A-4.5**) expressed in litres or kg; and

M = amount of the sample of the material or article expressed in g.

A-6.2 The concentration of VC in the material and article under investigation expressed in mg/kg shall be the average of the two concentrations of VC (mg/kg) determined under **A-6.l** provided that the repeatability criterion under **A-8** is satisfied.

A-7 CONFIRMATION OF THE VC LEVEL

A-7.1 In cases where the content of VC in materials and articles as calculated under **A-6.l** exceeds the maximum permissible amount the results obtained by the analysis of each of two samples (**A-5.1** and **A-6.1**) shall be confirmed in one of the three ways:

a) by using at least one other column (**A-3.3**) having a stationary phase with a different polarity. This procedure should continue until a chromatogram is obtained with no evidence of overlap of the VC and/or internal standard peaks with constituents of the sample of the material or article; or

b) by using other detectors, namely, the micro-electrolytic conductivity detector; or

c) by using mass-spectrometry. In this case, if molecular ions with parent masses (*m/e*) of 62 and 64 are found in the ratio of 3 : 1, it may be regarded with high probability as confirming the presence of VC. In case of doubt the total mass spectrum shall be checked.

A-8 REPEATABILITY

A-8.1 The difference between the results of two determinations (**A-6.1**) carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions shall not exceed 0.2 mg VC/kg of material or article.

ANNEX B

(*Clause* 4.4.2)

DETERMINATION OF VINYL CHLORIDE RELEASED BY MATERIALS AND ARTICLES INTO FOODSTUFFS (EEC METHOD)

B-1 GENERAL

This method determines the level of vinyl chloride in foodstuffs.

B-2 PRINCIPLE

The level of vinyl chloride (VC) in foodstuffs is determined by means of gas-chromatography using the 'headspace' method.

B-3 REAGENTS

B-3.1 Vinyl Chloride (VC), of purity greater than 99.5 percent (*v/v*).

B-3.2 N, N-Dimethylacetamide (DMA), free from any impurity with the same retention time as VC or as the internal standard (**B-3.3**) under the conditions of the test.

B-3.3 Diethyl Ether or cis-2-butene, in DMA (**B-3.2**) as the internal standard solution. These internal standards shall not contain any impurity with the same retention time as VC, under the conditions of the test.

B-3.4 Distilled Water or Demineralized Water of Equivalent Purity

B-4 APPARATUS

B-4.1 Gas-chromatograph fitted with automatic headspace sampler or with facilities for manual sample injection.

B-4.2 Flame ionization detector or other detectors mentioned in **B-7**.

B-4.3 Gas-Chromatographic Column

The column shall permit the separation of the peaks of air, of VC and of the internal standard, if used.

Furthermore, the combined **B-4.2** and **B-4.3** system shall allow the signal obtained with a solution containing 0.005 mg VC/litre DMA or 0.005 mg VC/kg DMA to be equal to at least five times the background noise.

B-4.4 Sample Phials or Flasks Fitted with Silicon or Butyl Rubber Septa

NOTE — When using manual sampling techniques, the taking of a sample from the heads pace with a syringe may cause a partial vacuum to form inside the phial or flask. Hence, for manual techniques where the phials are not pressurized before the sample is taken, the use of large phials is recommended.

B-4.5 Micro-Syringes

B-4.6 Gas-Tight Syringes for Manual Headspace Sampling

B-4.7 Analytical Balance Accurate to 0.1 mg

NOTE — An instrument or piece of apparatus is mentioned only if it is special, or made to particular specifications. Usual laboratory apparatus is assumed to be available.

B-5 PROCEDURE

CAUTION — VC is a hazardous substance and a gas at ambient temperature, therefore, the preparation of solutions shall be carried out in a well-ventilated fume cupboard.

NOTE — Take all the necessary precautions to ensure that no VC or DMA is lost. When employing manual sampling techniques, an internal standard (**B-3.3**) should be used. When using an internal standard, the same solution shall be utilized throughout the procedure.

B-5.1 Preparation of Standard VC Solution (Solution A)

B-5.1.1 *Concentrated Standard VC Solution at Approximately 2 000 mg/kg*

Accurately weigh to the nearest 0.1 mg a suitable glass vessel and place in it a quantity (50 ml) of DMA (**B-3.2**). Reweigh. Add to the DMA a quantity (0.1 g) of VC (**B-3.1**) in liquid or gas form, injecting it slowly onto the DMA. The VC may also be added by bubbling it into the DMA, provided that a device is used which will prevent loss of DMA. Reweigh to the nearest 0.1 mg. Wait for 2 h to allow equilibrium to be attained. If an internal standard is to be employed, add internal standard so that the concentration of the internal standard in the concentrated standard VC solution is the same as in the internal standard solution prepared under point **B-3.3**. Keep the standard solution in a refrigerator.

B-5.1.2 *Preparation of Dilute Standard VC Solution*

Take a weighed amount of concentrated standard solution of VC (**B-5.l.l**) and dilute, to a known volume or a known weight, with DMA (**B-3.2**) or with internal standard solution (**B-3.3**). The concentration of the resultant dilute standard solution (Solution A) is expressed as mg/l or mg/kg respectively.

B-5.1.3 *Preparation of the Response Curve with Solution A*

NOTE — The curve must comprise at least seven pairs of points. The repeatability of the responses shall below than0.002 mg VC/litre or kg of DMA. The curve shall be calculated from these points by the least squares technique that is the regression line must be calculated using the following equation:

$$\gamma = a_1 x + a_0$$

where

$$a_1 = \frac{n\sum xy - \left(\sum x\right).\left(\sum y\right)}{n\sum x^2 - \left(\sum x\right)^2}$$

and

$$a_0 = \frac{\left(\sum y\right)\left(\sum x^2\right) - \left(\sum x\right).\left(\sum xy\right)}{n\sum x^2 - \left(\sum x\right)^2}$$

where

y = the height or area of peaks in any single determination;

x = the corresponding concentration on the regression line; and

n = number of determinations carried out ($n \geq 14$).

The curve shall be linear, that is, the standard deviation(s) of the differences between the measured responses (y_i) and the corresponding value of the responses calculated from the regression line (z_i) divided by the mean value ($\bar{y}$) of all the measured responses shall not exceed 0.07.

This shall be calculated from:

$$\frac{s}{\bar{y}} \leq 0.07$$

where

$$S = \sqrt{\frac{\sum_{i=1}\left(y_i - z_i\right)^2}{n-1}}$$

and

$$\bar{y} = \frac{1}{n}\sum_{i=1}^{n} y_i$$

where

y_i = each individual measured response; and

z_i = the corresponding value of the response (y_i) on the calculated regression line, and $n \geq 14$.

Prepare two series of at least seven phials (**B-4.4**). Add to each phial volumes of dilute standard VC solution (**B-5.1.2**) and DMA (**B-3.2**) or internal standard solution in DMA (**B-3.3**) such that the final VC concentration of the duplicate solutions will be approximately equal to 0, 0.005, 0.010, 0.020, 0.030, 0.040, 0.050, etc mg/litre or mg/kg of DMA and that each phial contains the same total volume of solution. The quantity of dilute standard VC solution (**B-5.1.2**) shall be such that the ratio between the total volume (µl) of added VC solution and quantity (g or ml) of DMA, or internal standard solution (**B-3.3**) shall not exceed five. Seal the phials and proceed as described under **B-5.4.2, B-5.4.3** and **B-5.4.5**. Construct a graph in which the ordinate values show the areas (or heights) of the VC peaks of the duplicate solutions, or the ratio of these areas (or heights) to those of the relevant internal standard peaks, and the abscissa values show the VC concentrations of the duplicate solutions.

B-5.2 Validation of Preparation of Standard Solutions Obtained in B-5.1

B-5.2.1 *Preparation of a Second Standard VC Solution* (*Solution B)*

Repeat the procedure described under **B-5.1.1** and **B-5.1.2** to obtain a second dilute standard solution with, in this case, a concentration approximately equal to 0.02 mg VC/l or 0.02 mg VC/kg of DMA or internal standard solution. Add this solution to two phials (**B-4.4**). Seal the phials and proceed as described under **B-5.4.2, B-5.4.3**, and **B-5.4.5**.

B-5.2.2 *Validation of Solution A*

If the average of two gas-chromatographic determinations relating to the Solution B (**B-5.2.1**) does not differ by more than 5 percent from the corresponding point of the response curve obtained in **B-5.1.3**, the Solution A is validated. If the difference is greater that 5 percent, reject all the solutions obtained in **B-5.1**and **B-5.2** and repeat the procedure from the beginning.

B-5.3 Preparation of the 'Addition' Curve

NOTES

1 The curve shall comprise at least seven pairs of points.

2 The curve shall be calculated from these points by the least squares technique (**B-5.1.3**, third indent).

3 The curve shall be linear, that is, the standard deviation(s) of the differences between the measured responses (y) and the corresponding value of the responses calculated from the regression line (z_i) divided by the mean value (y) of all the measured responses shall not exceed 0.07 (**B-5.1.3**, fourth indent).

B-5.3.1 *Preparation of Sample*

The sample of foodstuff to be analyzed shall be representative of the foodstuff presented for analysis. The foodstuff shall, therefore, be mixed or reduced to small pieces and mixed before the sample is taken.

B-5.3.2 *Procedure*

Prepare two series of at least seven phials (**B-4.4**). Add to each phial a quantity, not less than 5 g of sample obtained from the foodstuff under investigation (**B-5.3.1**). Ensure that an equal quantity is added to each phial. Close the phial immediately. Add to each phial for each gram of sample 1 ml, preferably of distilled water, or demineralized water of at least equivalent purity, or an appropriate solvent if necessary.

NOTE — For homogenous foodstuffs, addition of distilled or demineralized water is not necessary.

Add to each phial volumes of dilute standard VC solution (**B-5.1.2**), containing the internal standard (**B-3.3**), if considered useful, such that concentration of VC added to the phials equal to 0.005, 0.010, 0.020, 0.030, 0.040 and 0.050, etc, mg/kg of foodstuff. Ensure that the total volume of DMA or DMA containing internal standard (**B-3.3**) in each phial is the same. The quantity of dilute standard VC solution (**B-5.1.2**) and additional DMA where used, shall be such that the ratio between the total volume (µl) of these solutions and the quantity (g) of foodstuff contained in the phial is as low as possible but not more than five and is the same in all phials. Seal the phials and proceed as described under **B-5.4**.

B-5.4 Gas-Chromatographic Determinations

B-5.4.1 Agitate the phials avoiding contact between the contained liquid and the septum (**B-4.4**) to obtain a solution or a suspension of the samples of foodstuff as homogenous as possible.

B-5.4.2 Put all the sealed phials (**B-5.2** and **B-5.3**) in a water bath for 2 h at 60 ± 1 ºC to allow equilibrium to be attained. Agitate again, if necessary.

B-5.4.3 Take a sample from the headspace in the phial. When utilizing manual sampling techniques care shall be exercised in obtaining a reproducible sample (**B-4.4**) in particular the syringe shall be pre-warmed to the temperature of the sample. Measure the area (or height) of the peaks relating to the VC and internal standard, if used.

B-5.4.4 Construct a graph in which the ordinate value shows the areas (or heights) of the VC peaks or the ratio of the areas (or heights) of VC peaks to the areas (or heights) of the internal standard peaks and the abscissa values show the quantities of VC added (mg) relating to the quantities of the sample of foodstuff weighed in each phial (kg). Measure the abscissa intercept from the graph. The value so obtained is the

concentration of VC in the sample of the foodstuff under investigation.

B-5.4.5 Remove from the column (**B-4.3**) excess DMA using a appropriate method as soon as peaks of DMA appear on the chromatogram.

B-6 RESULTS

The VC released by materials and articles into the foodstuff under the investigation expressed in mg/kg shall be defined as the average of the two determinations (**B-5.4**) provided that the repeatability criterion in **B-8** is satisfied.

B-7 CONFIRMATION OF THE VC

In cases where the VC released by materials and articles into the foodstuffs as calculated under **B-6**, exceeds the criteria in Article 2 (2) of Council Directive 78/142/EEC of 30 January 1978, the values obtained in each of the two determinations (**B-5.4**) shall be confirmed in one of the three ways:

a) by using at least one other column (**B-4.3**) having a stationary phase of different polarity. This procedure shall continue until chromatogram is obtained with no evidence of overlap of the VC and/or internal standard peaks with constituents of the sample of foodstuff;

b) by using other detectors, for example, the micro-electrolytic conductivity detector; and

c) by using mass spectrometry; in this case, if molecular ions with parent masses (*m*/*e*) of 62 and 64 are found in the ratio of 3 : 1 it may be regarded with high probability as confirming the presence of VC. In case of doubt the total mass spectrum shall be checked.

B-8 REPEATABILITY

The difference between the results of two determinations (**B-5.4**) carried out simultaneously or in rapid succession on the same sample, by the same analyst, under the same conditions, shall not exceed 0.003 mg VC/kg of foodstuff.

ANNEX C

(*Clause* 4.5.1)

METHOD OF PREPARATION OF FILM/SHEET FROM PVC RESIN FOR TESTING OVERALL MIGRATION

C-1 SCOPE

This Annex describes the method for preparation of film/sheet from PVC resin for overall migration testing.

C-2 TERMINOLOGY

C-2.1 Friction Ratio — Ratio of the speed of the fast to the slow roll.

C-3 APPARATUS

C-3.1 Mixing Mill — The laboratory mixing mill has two parallel cylindrical hardened steel rolls 150 ± 5 mm in outside diameter. The rolls are fitted with adjustable guides to allow a maximum working width of 26.5 ± 1.5 cm. The mill has provision for maintaining the temperature of the roll surfaces at 170° to 180°C during the mixing. The two rolls rotate at different speeds. The speed of the slow roll is 24 ± 1 rev/min and the friction ratio 1 : 1.4.

C-3.1.1 If mills having ratios of fast to slow roll speeds lower than 1 : 1.4 are used, modifications in the mixing conditions given under procedure may be required to obtain results comparable to those obtained with the standard mill.

C-3.1.2 The mill is designed to permit adjustment of the distance between the rolls from 0.2 mm or less, to 3.0 mm or more.

C-3.2 Surface Pyrometer or Other Device — To determine the temperature of the roll surfaces accurate to within 0.5 °C.

C-4 STANDARD COMPOUND

C-4.1 Ingredients

Only materials of PVC grade quality shall be used.

C-4.2 Composition

The standard film/sheet shall have the following formulation :

	Parts by Mass
Polyvinyl chloride (PVC)	100
Epoxidized oil (Soyabean)	1.0
Di-*n*-octyl (tin s-s'-bis*iso*-octylmercapto- acetate)	1.5
Stearic acid	0.8

C-4.3 All ingredients shall be kept in closed containers at the standard laboratory temperature 27 ± 2°C for a period of not less than 15 h and shall not be exposed to the air for longer than is necessary.

C-5 MIXING PROCEDURE

C-5.1 Batch Size

The batch size may be either same or twice the parts by mass given in **C-4.2**.

C-5.2 Weighing of Ingredients

All ingredients shall be weighed to within one percent of the specified mass. The mass of the mixed batch shall not differ from the mass of all the ingredients by an amount exceeding 0·5 percent. The ingredients shall be weighed separately for each batch, except when it is agreed that master-batches may be used.

C-5.3 Preparation of Film/Sheet

C-5.3.1 The mixing temperature is 170 to 180 °C.

C-5.3.2 Weigh the ingredients and mix in a beaker with spatula.

C-5.3.3 Put the mixture of all ingredients on two roll mill. Adjust the distance between the rolls to get film of requisite thickness. Measure the thickness, using an accurate micrometer or thickness gauge. Continue milling for 10 min. Remove the sheet and cut requisite size filmfor determining overall migration in accordance with IS 9845.

ANNEX D

(*Clause* 6.1)

SAMPLING OF PVC AND ITS COPOLYMERS

D-1.1 In drawing, preparing, storing and handling samples, the following precautions and directions shall be observed.

D-1.2 Samples shall not be taken in an exposed place.

D-1.3 The sampling instrument, wherever applicable, shall be made of stainless steel or any other suitable material on which the material shall have no action. The instrument shall be clean and dry.

D-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

D-1.5 The samples shall be placed in a suitable, clean, dry, air-tight metal or glass containers on which the material has no action. The sample containers shall be of such a size that they are almost completely filled by the sample.

D-1.6 Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sampling, such as the date of sampling, the month and year of manufacture of the material, etc.

D-1.7 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

D-2 SCALE OF SAMPLING

D-2.1 Lot

In a single consignment all the packages of the same class, same type, same form and belonging to the same batch of manufacture shall be grouped together to constitute a lot. If a consignment is known to consist of packages belonging to different batches of manufacture or different forms, the packages belonging to the same batch of manufacture and same form shall be grouped together and each such group shall constitute a lot.

D-2.1.1 The packages may consist of containers of PVC and its copolymers, rolls, films or vials.

D-2.2 For ascertaining the conformity of the material to the requirements of this specification, samples shall be tested from each lot separately. The number of packages to be sampled shall depend on the size of the lot and shall be in accordance with col 1 and 2 of Table 1.

D-2.2.1 These packages shall be selected at random from the lot and in order to ensure the randomness of selection, procedures given in IS 4905 may be followed.

Table 1 Scale of Sampling
(*Clause* D-2.2)

Number of Packages in the Lot (1)	Sample Size (2)
Up to 15	2
16 to 50	3
51 to 100	4
101 to 300	5
301 to 500	6
501 to 1 000	8
1 001 and above	10

NOTE — When the number of packages in the lot is less than three, all the packages shall be sampled.

D-3 PREPARATION OF TEST SAMPLES

D-3.1 From each of the packages of material selected, small portions of material shall be drawn with the help of a suitable sampling instrument. The total quantity of material collected from each package shall be sufficient to test all the requirements given in **3** of the standard.

D-3.2 In the case of packages consisting of containers, vials, rolls or films, the number of items to be selected from a package for testing each of the requirements given in **4** of the standard, shall be one.

D-4 NUMBER OF TESTS

D-4.1 Tests for determining all the requirements given in **4** of the standard shall be carried out on the individual test samples.

D-5 CRITERIA FOR CONFORMITY

D-5.1 The lot shall be declared as conforming to the requirements of this specification if all the test results on individual samples meet the relevant specification requirements.

ANNEX E

(*Foreword*)

LIST OF INDIAN STANDARDS ON PLASTICS SUITABLE FOR USE IN CONTACT WITH FOODSTUFFS, PHARMACEUTICALS AND DRINKING WATER

IS No.	*Title*
9833 : 2014	List of pigments and colorants for use in plastics in contact with foodstuffs, Pharmaceuticals and drinking water (*first revision*)
9845 : 1998	Determination of overall migration of constituents of plastics materials and articles intended to come in contact with foodstuffs — Method of analysis (*second revision*)
10141 : 2001	Positive list of constituents of polyethylene in contact with foodstuffs, pharmaceuticals and drinking water (*first revision*)
10142 : 1999	Polystyrene (crystal and high impact) for its safe use in contact with foodstuffs, pharmaceuticals and drinking water — specification (*first revision*)
10146 : 1982	Polyethylene for its safe use in contact with foodstuffs, pharmaceuticals and drinking water
10148 : 1982	Positive list of constituents of polyvinyl chloride and its copolymers for safe use in contact with foodstuffs, pharmaceuticals and drinking water
10149 : 1982	Positive list of constituent of styrene polymers in contact with foodstuffs, pharmaceuticals and drinking water
10151 : 1982	Polyvinyl chloride (PVC) and its copolymers for its safe use in contact

IS No.	*Title*
	with foodstuffs, pharmaceuticals and drinking water
10171 : 1999	Guide on suitability of plastics for food packaging (*second revision*)
10909 : 1984	Positive list of constituents polypropylene and its copolymers for its safe use in contact with foodstuffs, pharmaceuticals and drinking water (*first revision)*
10910 : 1984	Specification for polypropylene and its copolymers for its safe use in contact with foodstuffs, pharmaceuticals and drinking water
11434 : 1985	Ionomer resins for its safe use in contact with foodstuffs, pharmaceutical and drinking water
11435 : 1985	Positive list of constituents of ionomer resins for its safe use in contact with foodstuffs, pharmaceuticals and drinking water
11704 : 1986	Ethylene/acrylic acid (EAA) copolymers for their safe use in contact with foodstuffs, pharmaceuticals and drinking water
11705 : 1986	Positive list of constituents of ethylene/acrylic acid (EAA) Copolymers for their safe use in contact with foodstuffs, pharmaceuticals and drinking water
12229 : 1987	Positive list of constituents of Poly alkylene terephthalates (PET and PBT) for their safe use in contact with foodstuffs, pharmaceutical and drinking water
12247 : 1988	Nylon-6 polymer for its safe use in contact with foodstuffs pharmaceuticals and drinking water
12248 : 1988	Positive list of constituents of nylon-6 polymer for its safe use in contact with foodstuffs, pharmaceuticals and drinking water
12252 : 1987	Polyalkylene terephthalates (PET and PBT) for their safe use in contact with foodstuffs, pharmaceuticals and drinking water
13449 : 1992	Positive list of constituents of ethylene vinyl acetate (EVA) copolymers in contact with foodstuffs, pharmaceuticals and drinking water
13576 : 1992	Ethylene menthacrylic acid (EMAA) copolymers and terpolymers for their safe use contact with foodstuffs, pharmaceuticals and drinking water
13577 : 1992	Positive list of constituents of ethylene methacrylic acid (EMAA) copolymers and terpolymers in contact with foodstuffs, pharmaceuticals and drinking water
13601 : 1993	Ethylene vinyl acetate (EVA) copolymers for its safe use in contact with foodstuffs, pharmaceuticals and drinking water
14971 : 2001	Polycarbonate resins for its safe use in contact with foodstuffs, pharmaceuticals and drinking water
14972 : 2001	Positive list of constituents of polycarbonate resins in contact with foodstuffs, pharmaceuticals and drinking water
14996 : 2001	Positive list of constituents of modified poly (phenylene oxide) (PPO) in contact with foodstuffs, pharmaceuticals and drinking water
14997 : 2001	Modified poly (Phenylene oxide) (PPO) for their safe use in contact with foodstuffs, pharmaceuticals and drinking water
14998 : 2001	Positive list of constituents of melamine- formaldehyde resins in contact with foodstuffs, pharmaceuticals and drinking water
14999 : 2001	Melamine-formaldehyde moulding materials for its safe use in contact with foodstuffs, pharmaceuticals and drinking water

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 2016 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards : Monthly Additions'.

This Indian Standard has been developed from Doc No.: PCD 12 (2683).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

BUREAU OF INDIAN STANDARDS

Headquarters:

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 2323 0131, 2323 3375, 2323 9402 *Website*: www.bis.org.in

Regional Offices: *Telephones*

Central : Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002 — 2323 7617, 2323 3841

Eastern : 1/14 C.I.T. Scheme VII M, V. I. P. Road, Kankurgachi, KOLKATA 700054 — 2337 8499, 2337 8561, 2337 8626, 2337 9120

Northern : Plot No. 4-A, Sector 27-B, Madhya Marg, CHANDIGARH 160019 — 26 50206, 265 0290

Southern : C.I.T. Campus, IV Cross Road, CHENNAI 600113 — 2254 1216, 2254 1442, 2254 2519, 2254 2315

Western : Manakalaya, E9 MIDC, Marol, Andheri (East), MUMBAI 400093 — 2832 9295, 2832 7858, 2832 7891, 2832 7892

Branches: AHMEDABAD. BENGALURU. BHOPAL. BHUBANESHWAR. COIMBATORE. DEHRADUN. DURGAPUR. FARIDABAD. GHAZIABAD. GUWAHATI. HYDERABAD. JAIPUR. JAMSHEDPUR. KOCHI. LUCKNOW. NAGPUR. PARWANOO. PATNA. PUNE. RAIPUR. RAJKOT. VISAKHAPATNAM.

Published by BIS, New Delhi